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FLOW AND FRACTURE IN SPINEL STRUCTURED CERAMICS

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TECHNICAL REPORT NO. 1

"MULTIPLE SLIP PROCESSES IN MAGNESIUM ALUMINATE AT HIGH TEMPERATURES"

by

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August, 1965

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"MULTIPLE SLIP PROCESSES IN MAGNESIUM ALUMINATE AT HIGH TEMPERATURES"

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ABSTRACT

Evidences obtained from high purity $MgAl_2O_4$ ceramics deformed at high temperatures are presented which confirm theoretically predicted multiple (111)[110] slip systems for spinel. Stress-strain and kinetic data and microstructural examinations indicate that spinel, when sufficiently pure, does meet the Taylor-von Mises criterion for generalized plastic flow of a polycrystalline solid.

INTRODUCTION

A paper presented in the Special Ceramics Symposium at Stoke-on-Trent in June 1964[1] described N. C. State University's interest in spinel. It placed special emphasis on variations in strength and other mechanical properties of polycrystalline magnesium aluminate which can be attributed to the composition and homogeniety of starting materials, fabrication history, and resultant microstructure. Much of that prior paper was, in face, converned with efforts to develop and characterise "well behaved" polycrystalline spinel ceramics, so that

research on high temperature flow and fracture could be carried out under controlled conditions. Such studies with polycrystalline spinel, and parallel ones with single crystals, are now sufficiently advanced to provide the basis of this report of ductility in fine grained spinel ceramics at temperatures above approximately 0.7 T_M.

DEFORMATION IN CERAMICS

If structural integrity is to be preserved, grains within a glass-free polycrystalline solid must be able to undergo progressive changes in shape during deformation. If grains cannot deform, then translation, rotation and separation of individual grains must inevitably occur as strain progresses. Most crystalline ceramics cannot be permanently deformed by grain boundary sliding alone, even at high temperatures, without progressive grain boundary separation and eventual gross fracture.[2,3,4] The bulk volume must increase as separations form along the boundaries, and of course, any long narrow void in a boundary becomes mechanically equivalent to a propagatable crack under an appropriate applied stress.[5]

Two quite different mechanisms may be available at high temperature for accomplishing generalized changes of grain shape in crystalline ceramics, thus preserving structural integrity. In the first example, enhanced ionic mobility at elevated temperatures leads to diffusion processes capable of achieving mass transport from regions under compression toward tension regions of the stress field. Such

mechanisms form the basis of the well known Nabarro-Herring process for creep.[6, 7] Diffusional creep is mathematically described as a viscous process; it involves low stress, low strain rates, and a linear strain rate-stress relationship. In creamics, such creep processes usually occur at strain rates measured in very small fractions of inches per inch per hour.[7]

The second flow process by which structural integrity can be preserved during deformation involves crystalline plasticity. For plasticity, mobility of dislocations is a first requirement, one which can be attained in most crystalline solids by sufficient thermal activation. In single crystal form, anisotropic refractory oxides and even covalent-bonded carbides can demonstrate considerable high temperature ductility in orientations favorable for slip of dislocations.[8]

In a polycrystalline solid, however, constraints to slip are developed between neighboring grains having randomly different orientations; high angle boundaries between such grains act as very effective barriers to oncoming dislocations. In such a situation, thermally activated dislocation mobility alone is not sufficient for constant volume plastic flow, but there also must be a sufficient number of independent slip systems to achieve a generalized and self-contained change in grain shape. This limiting geometric condition is expressed in the Taylor-Von Mises criterion,[4, 9, 10] which specifies that at least five independent systems are required for polycrystalline plasticity. According to Groves and Kelly,[11] none of the common

ceramic materials meet this criterion at low temperatures. In a very recent paper, Pratt et al demonstrated plastic behavior in polycrystalline CaF_2 at temperatures as low as 350°C.[12] Even at high temperatures, only those ceramics having NaCl and CaF_2 structures have usually been considered capable of developing ductility in polycrystalline form.[4,13]

Chang[14] has described steady state creep in plastically deforming ceramic solids at some high temperature as that stress-strain rate condition where the rate of work hardening due to pileup of dislocations at slip bands, grain boundaries and other obstacles is exactly matched by the rate of recovery, i.e., the rate at which dislocation "debris" can be dissipated at that temperature by diffusional processes. The earlier Weertman[15] analysis for creep invoked another diffusional process, climb of dislocations into unused slip planes, as the rate controlling step.

Plastic creep in polycrystalline solids is characterized by steep logarithmic strain rate-stress slopes (generally on the order of 3 to 6), and by an activation energy higher than that for plastic flow in a single crystal. Its magnitude is usually on the order of the activation energy for self-diffusion for the slow moving ionic or atomic species. Under favorable temperature-stress conditions, strain rates in the fractional inch per inch per minute range are typical for steady state plastic flow, and for metals, may be one or more orders of magnitude higher.

DEFORMATION IN SPINEL

The spinel structure is almost unique among ceramic crystal types in that its dislocations are arranged somewhat like those of a face centered cubic metal. Its structure is complex, but the (111) slip planes are anion close-packed planes, and [110] oxygen-oxygen directions are slip directions.[16, 18] These multiple (111)[110] slip systems are schematically illustrated for uniaxial loading along [110], [111], and [100] in Figures 1, 2, and 3 respectively.

In the [110] case, two slip planes are equally oriented to receive shear stress, and each has two equally stressed Burgers vectors along which slip can occur; these two planes contain four independent slip systems. The two remaining planes are parallel to the load axis so that dislocations lying in them are subject to normal rather than shear stresses, and hence should not glide. This orientation does, however, present a classical opportunity for a special type of dislocation motion called kinking to occur.[17]

When the load axis coincides with [111], as in Figure 2, there are three equally oriented planes subject to shear, and each as two operative Burgers vectors. In this case, six independent systems are evident.

For the [100] load direction (Fig. 3), four equally oriented (111) planes are in shear geometry, and each has two favorably oriented Burgers vector directions. In this direction, eight independent slip

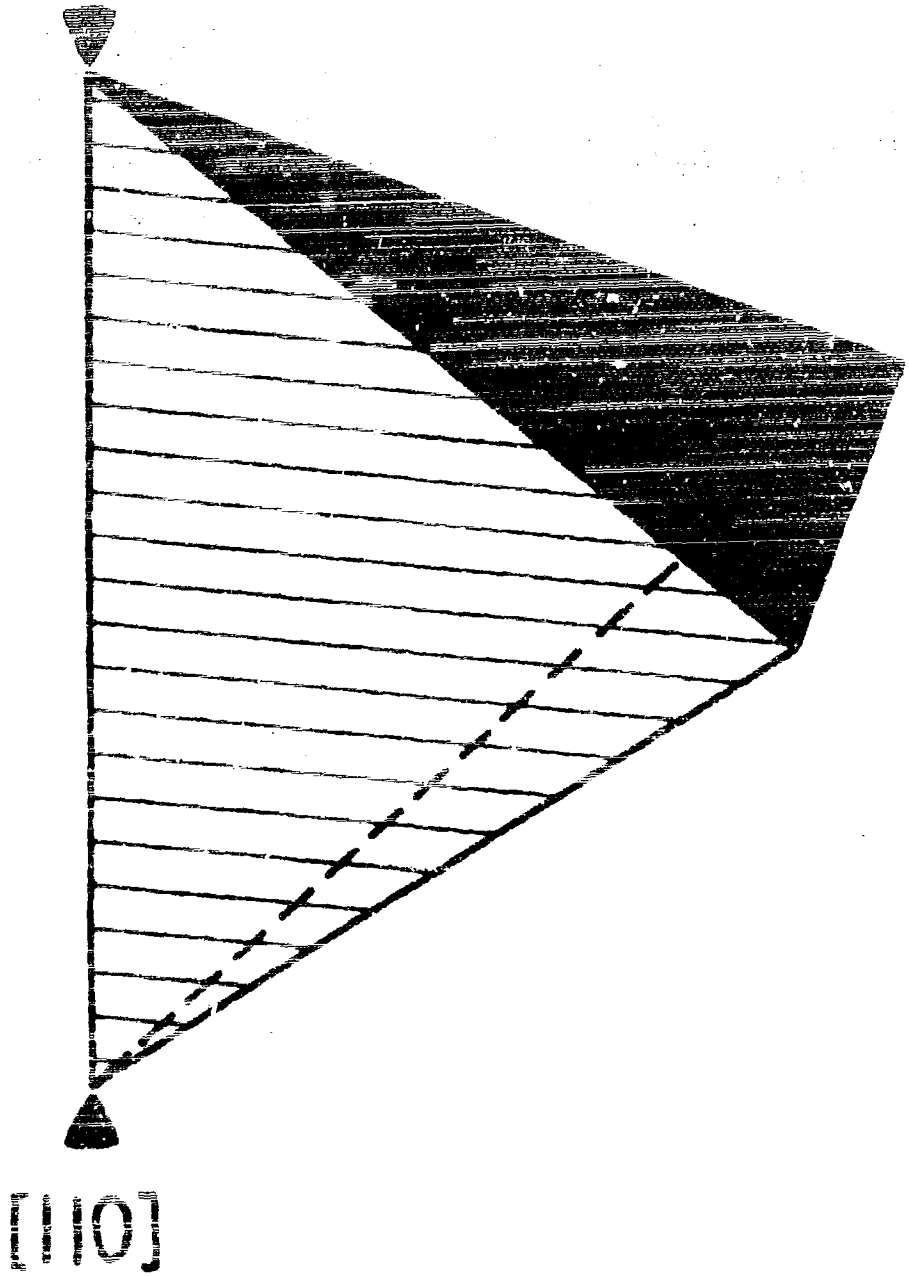

Figure 1. Geometry for Slip in Spinel, Uniaxial Normal Stress along [110]. Upper and lower triangles each have two equivalent operative slip systems with Burgers vectors perpendicular to each of the converging sides. Front and back triangles receive only normal stress; dislocations on them are immoble unless local misorientation or lateral shear initiates kinking. Four systems operative.

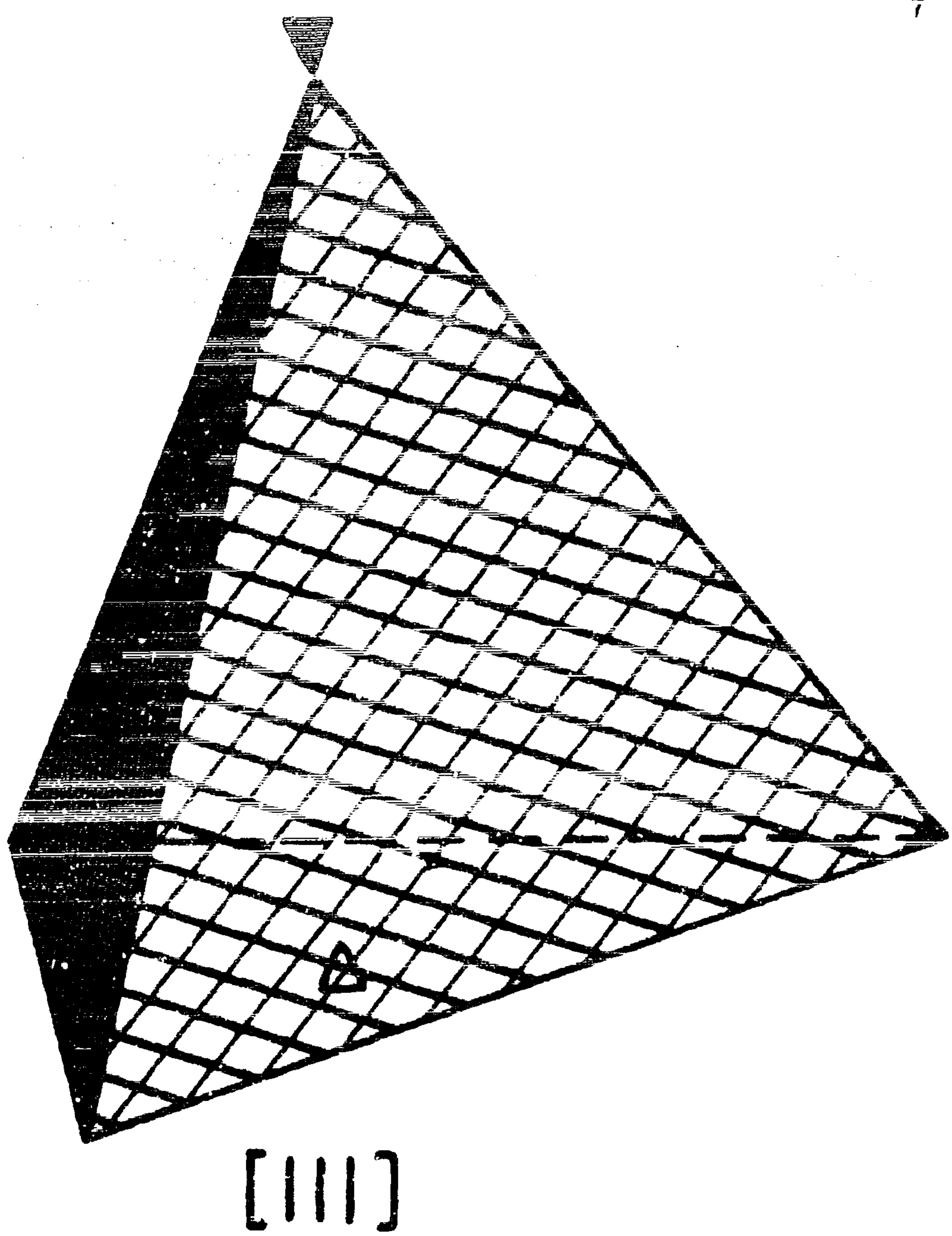

Figure 2. Geometry for Slip in Spinel, Uniaxial Normal Stress along [111]. Three triangles converging at apex each have two equivalent operative slip systems with Burgers vectors parallel to each of the converging sides. Bottom triangle receives only normal stress. Six systems operative.

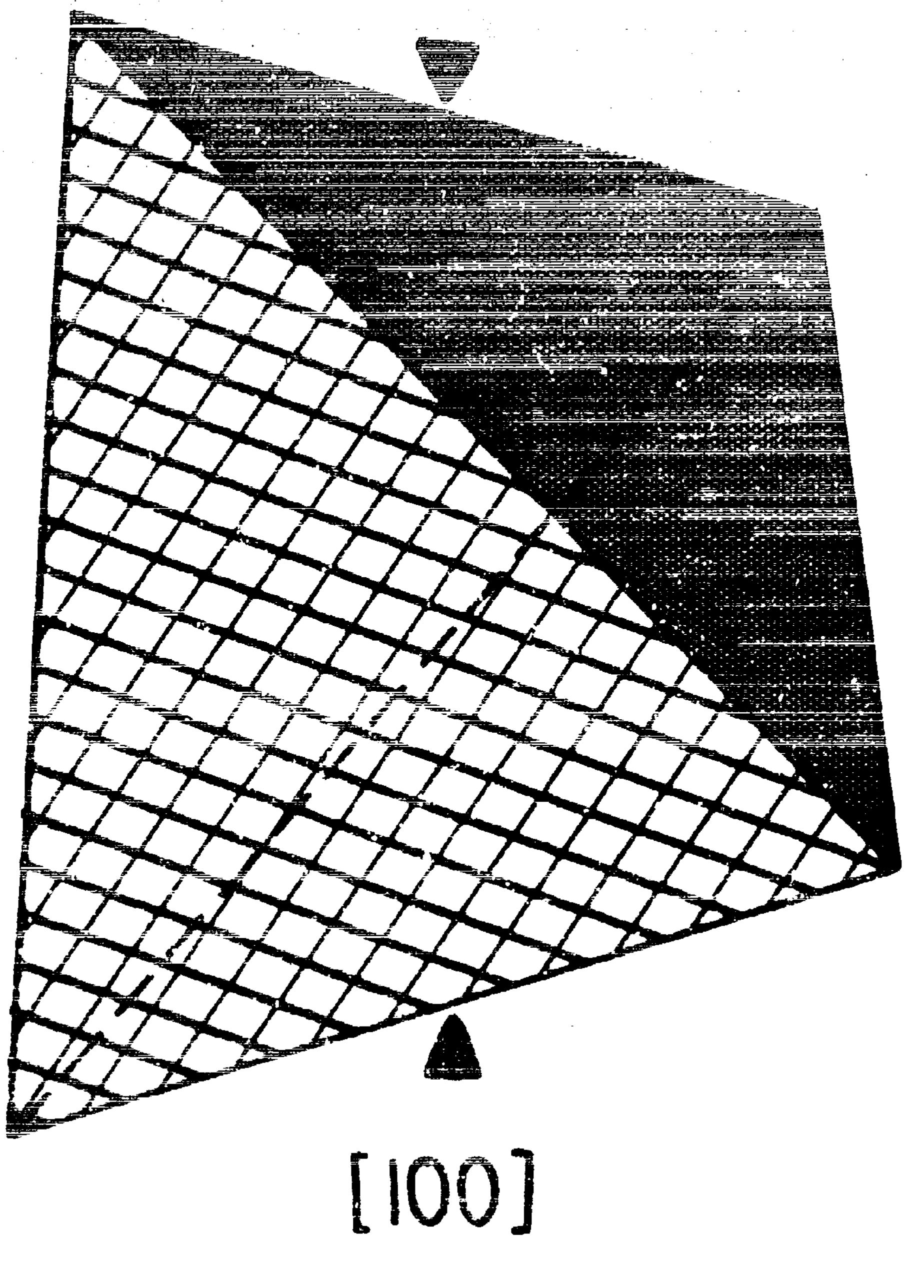

Figure 5. Geometry for Slip in Spinel, Uniaxial Normal Stress along [100]. All four triangles receive shear stress, and each has two equivalent operative slip systems. Eight systems operative.

systems seem assured.

Table 1 summarizes operative slip systems and resolved shear stresses and strains for uniaxial stress applied parallel to these low index directions in spinel.

Compression of Single Crystals

Spinel crystals uniaxially deformed about 4% in compression parallel to [110], [111], and [100] directions at temperatures above 1550°C at strain rates between 0.001 min^{-1} and 0.1 min^{-1} demonstrate just such behavior.* The morphology after deformation is entirely consistent with that predicted from these geometrical considerations of slip, even to the consistently observed broadening of the ($\bar{1}10$) face by interpenetrating slip and the concurrent kinking visible on the orthogonal (010) face for crystals stressed on [110] when misorientations of as little as 5°, or accidentally applied shear stresses, have been present.

Single crystal studies are being carried out in our laboratory with specimens cut from one large boule of an alumina-rich spinel solid solution (Al_2O_3 : MgO 2.9 : 1). Spinel crystals with 1 : 1 stoichiometry of a size and quality suitable for such deformation studies have not been successfully synthesized.[18] Consequently, these deformation studies are being carried out above 1550°C (approached at a

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*R. Douglas McBrayer, Doctoral Dissertation. To be published.

TABLE I. ORIENTATION DEPENDENCE OF PLASTIC FLOW IN SPINEL

Load Axis	No. Planes	No. Slip Systems	ϕ	λ	Resolved* Shear Stress τ	Resolved** Shear Strain γ
[100]	4	8	54.7°	45°	$0.4086\,\sigma$	$1.414\,\varepsilon$
[110]	2	4	35.3°	60°	$0.4081\,\sigma$	$2\,\varepsilon$
	2	Kinking	$\sim 90^\circ$	0°	$\sim 0.0\,\sigma$	ε
[111]	3	6	70.5°	35.3°	$0.2724\,\sigma$	$1.225\,\varepsilon$

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* $\tau = (\cos\phi \cos\lambda)\,\sigma$

** $\gamma = \dfrac{\varepsilon}{\cos\lambda}$

rate of $50^{\circ}C\ min^{-1}$) to avoid exsolution of hyperstoichiometric alumina as a second phase. Rapid cooling of the furnace after completion of a test does not entirely eliminate exsolution at surface discontinuities remanent from grinding and polishing.

A typical stress-strain plot for one such single crystal specimen is illustrated in Figure 4. The yield stress was very low, and the region up to 3% strain (note compressed scale) is attributed to work hardening. It was followed by a steady state creep region at a flow stress of 8000 psi, which continued until the specimen was unloaded after 5% strain. Temperature fluctuations are responsible for stress deviations in the steady state region.

Compression of Polycrystalline Spinel

A comparable study of high temperature deformation in polycrystalline spinel has also been carried out, and will form the basis of a subsequent paper.* Careful microstructural examinations by fractography,[20] and, more recently, of thermally and/or chemically etched specimens deformed under various conditions, have yielded conclusive evidences of truly plastic behavior in polycrystalline spinel. Coarse grained specimens ($d \simeq 200\ \mu$) in particular are rich with examples of wavy slip, localized recrystallization, generation of grain boundary separations and Stroh-Petch[21] cracks (traversing the boundaries) in heavily worked regions. Figure 4 illustrates the marked

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*Dong M. Choi, Doctoral Dissertation. To be published.

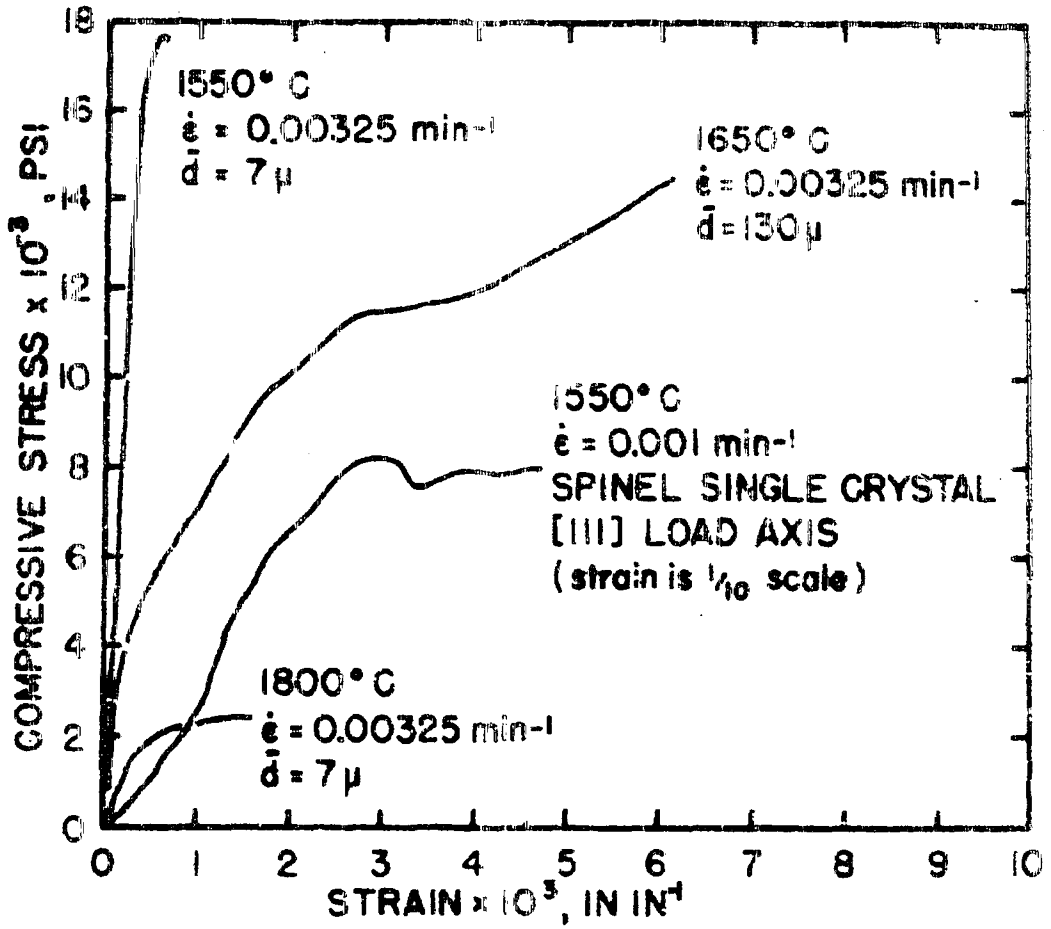

Figure 4. Stress-Strain Diagrams for Single Crystal and Polycrystalline Spinel. Specimens deformed in compression above 1550°C.

effect of temperature and grain size upon typical compressive stress-strain diagrams obtained from dense polycrystalline spinel specimens. A very strong strain rate dependence is also found ($\dot{\varepsilon} \simeq \sigma^n$, where n ranges from 3 to 7 depending on grain size temperatures). All these results strongly suggest that polycrystalline spinel does deform by crystalline plasticity, a process involving mobility of dislocations, at temperatures above about 1300°C.

Kinetic Considerations of Flow in Dense Polycrystalline Spinel

As a working model for the flow process in polycrystalline spinel a phenomenological strain-rate equation is proposed which is analogous to the Dorn[22] creep rate expression but with the addition of an expression (d^{-m}) to describe the effect of grain size:

$$\dot{\varepsilon} = K\sigma^{n} e^{-Q/RT} d^{-m} \qquad (1)$$

In logarithmic form, and with the dependent experimental variable (σ, the flow stress) transposed to the left hand member, the simplest expression becomes:

$$\ln\sigma = -\left(\frac{1}{n}\right) \ln K + \left(\frac{1}{n}\right) \ln \dot{\varepsilon} + \left(\frac{Q}{nR}\right) \frac{1}{T} + \left(\frac{m}{n}\right) \ln d \qquad (2)$$

Since there is a considerable likelihood of higher order effect, and particularly of interactions between variables (e.g., a temperature-grain size interaction influencing flow stress might come about if grain size or grain shape is altered as a function of the test temperature), the high temperature compression experiments have been

designed for analysis in terms of the expanded Taylor's series model commonly used in data reduction by statistical methods:[23, 24]

$$Y = b_0 + b_1X_1 + b_2X_1^2 + b_3X_2 + b_4X_2^2 + b_5X_3 + b_6X_3^2 \tag{3}$$
$$+ b_7X_1X_2 + b_8X_1X_3 + b_9X_2X_3$$

where

$$X_1 = f\ (\ln \dot{\varepsilon})\quad 0.0005\ \mathrm{min}^{-1}\ \ \dot{\varepsilon}\ \ 0.05\ \mathrm{min}^{-1}$$

$$X_2 = f\ \frac{1}{T}\quad 1626\ ^{\circ}K\quad T\quad 2073\ ^{\circ}K$$

$$X_3 = f\ (\ln d)\quad 0.5\quad d\quad 200$$

If the coefficients of the quadratic and interaction terms should be insignificant, then the simplest model (Eq. 2) will have been shown to be valid, and

$$Y = \ln \sigma$$
$$b_0 = \frac{-\ln K}{n}$$
$$b_1 = \frac{1}{n} \tag{4}$$
$$b_3 = \frac{Q}{nR}$$
$$b_5 = \frac{-m}{n}$$

thus providing the quantities K, n, Q, and m which will serve to describe the kinetics of the process. However, should any of the quadratic (e.g., X_2^2) or interaction terms (e.g., X_1X_2) prove to be statistically significant, the simplest model cannot be justifiably employed, and it will be necessary to develop a more sophisticated interpretation of the flow process.

Bending of Polycrystalline Spinel

In recent experiments, polycrystalline spinel also has been deformed in transverse bending at temperatures ranging from 1450°C to 1700°C. These tests were carried out in a vacuum furnace* operating at 2×10^{-5} torr, as were the compression studies already described. The furnace is fitted on an Instron physical testing machine,** as shown in Figure 5. The combined assembly and its associated instrumentation provide measurement, control, and/or programming capability over temperature (to 2500°C), strain rate (2 in. min^{-1} and downward by about five orders of magnitude) and load (sensitivity from 1 to 10,000 pounds full scale). Stabilized water pressure is provided to eliminate fluctuations in applied stress attributable to length changes in the water-cooled load column. Much care is exercised in achieving axial alignment, and it is also necessary to correct the load weighing system for the pull of the vacuum on the upper column which links the sample to the load cell. When these precautions have been taken, the uncertainty in measuring applied load is on the order of one pound or less, and is attributable principally to the frictional drag of O-ring seals through which the load columns must move.

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*Model 1064 vacuum furnace, a product of the Richard Brew Co., Concord, N. H.

**Model TTCLM, a product of Instron Engineering Corp., Canton, Mass.

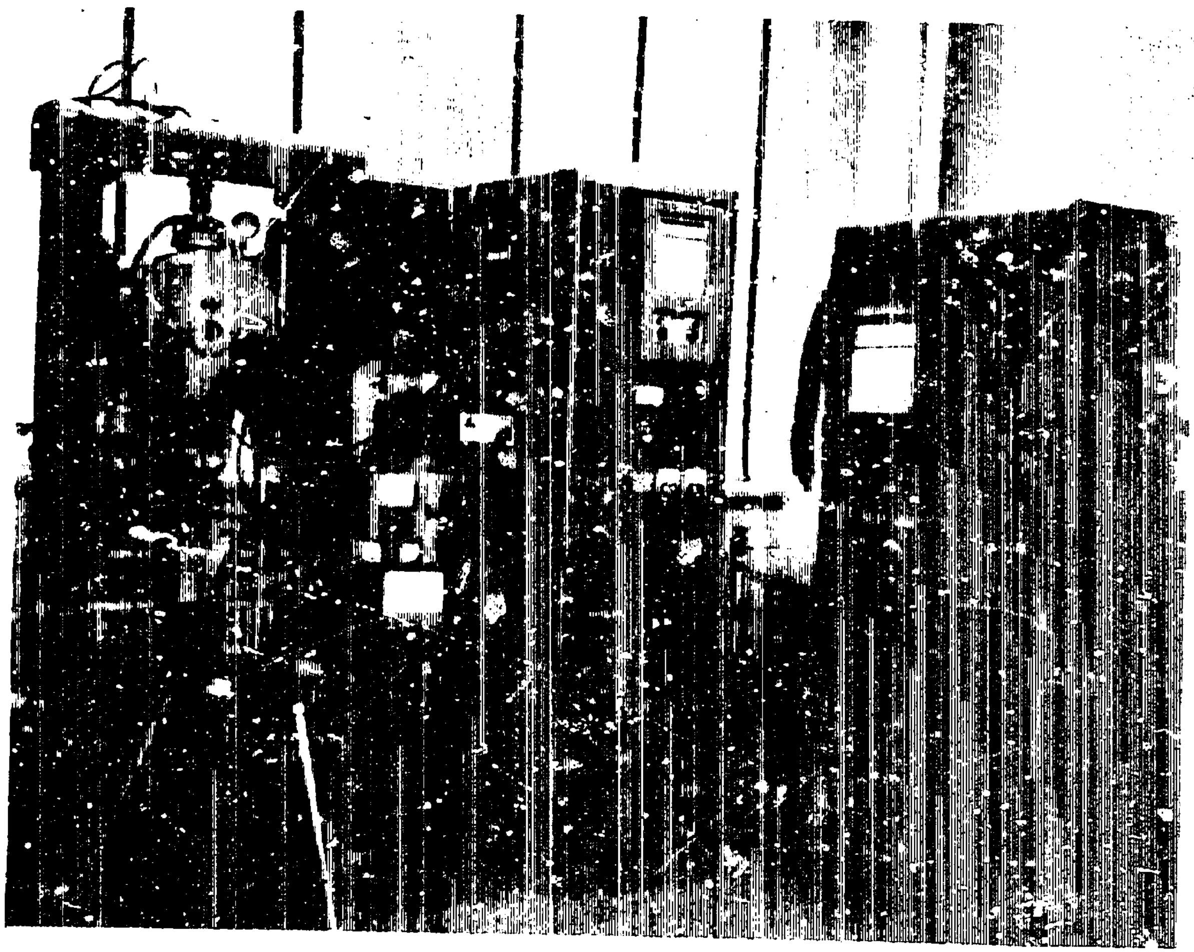
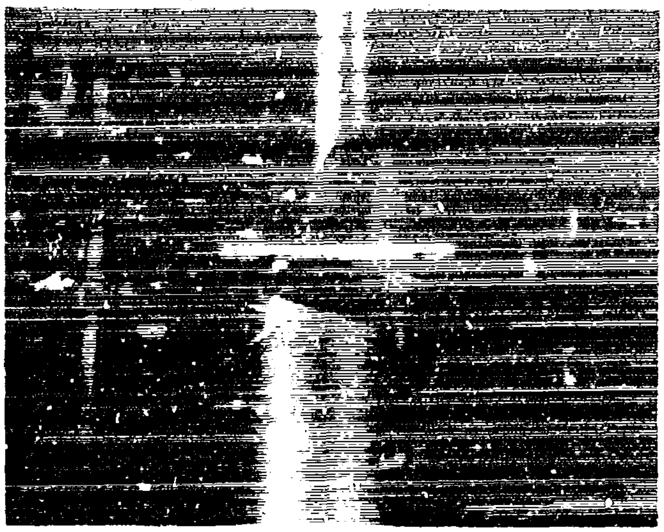


Figure 5. High Vacuum, High Temperature Physical Testing Facility (Instron-Brew).

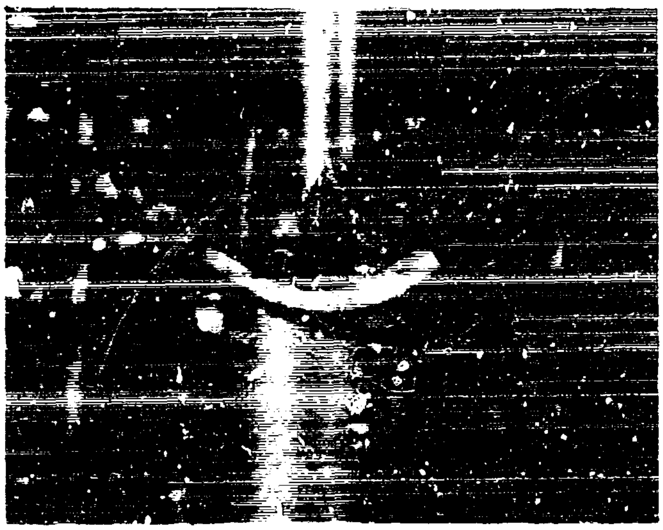
For these four-point bending experiments, a molybdenum fixture (shown in Fig. 6) was employed. It provides a 1.000" span on the lower block, which is articulated on an internal ball and socket joint in such a way that it can adjust to evenly distribute load on the knife edges by tilting slightly fore and aft, and even more in the picture plane. However, it is pinned to prohibit rotation by more than a few degrees about the load axis. The upper push rod has a span of 0.500", and is fixed in the machine in proper relationship with the lower block by use of an aligning jig. The 90° knife edges are rotated 15° from the vertical, and are dressed to provide a slight radius.

The specimens used were cut from a single piece of hot pressed spinel of >99.95% purity. Its average grain size was approximately 2μ, and its density was 97.4% of theoretical (based on 3.60 g/cc). The individual specimens were cut with a diamond saw, then ground flat and parallel on the beam faces to within ± 0.001". Their nominal size was 0.090" x 0.360" x 1.30". Retainers of 0.010" dia. molybdenum wire were employed to keep the specimens in place on the bending jig.

After inserting the specimen and putting the furnace under vacuum, a small stress was applied and removed several times to establish the point of zero stress, and to reference strain recording micrometers at the point of contact. The block was then lowered by an amount sufficient to insure that thermal expansion of the molybdenum load columns did not put the specimen under stress prior to the actual



(a)



(b)

Figure 6. Transverse Bending of Polycrystalline Spinel.
(a) Specimen in position prior to test.
(b) Specimen bent at 1700°C to 5% tensile at a nominal strain rate of 0.01 min^{-1}. Four-point molybdenum bending jig, rear tantalum heating element are visible.

test. Heating at approximately $50^{\circ}C$/min was programmed, using a W-W 26% Re thermocouple input. A two-color pyrometer,* essentially free from emissivity effects, was used to record and control at the test temperature, and a disappearing filament optical pyrometer was employed for an independent temperature check. Measurement and control under these conditions is considered to be reliable within $\pm 10^{\circ}C$ in the absolute sense when sighting on a black body; very small changes in temperature ($\approx 1^{\circ}C$) in the relative sense can be detected and corrected. The semicolloidal carbon "Prodag" intended to provide a painted-on black body sighting spot in this experiment persistently diffused into the molybdenum, leaving a fairly bright molybdenum carbide surface, so that reflectivity from the hotter heating elements was not entirely excluded. Hence the indicated temperatures may have been somewhat higher than the specimens actually attained.

Figure 6b illustrates a specimen after approximately 5% outer fiber tensile strain, accomplished at $1700^{\circ}C$ at strain rates which ranged from 0.00125 min^{-1} to 0.01 min^{-1}. The flow stress with the latter strain rate was about 200 psi, with a very slight upward trend indicative of work hardening. At the conclusion of the test, the specimen was intact, with every indication of full retention of structural integrity.

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*Coloratio Pyrometer, Latronics Corp., Latrobe, Pa.

Figure 7 illustrates stress-strain relationships for this and some other specimens deformed in bending. There is marked temperature and strain rate dependence, and considerable evidence of work hardening at the lower temperatures and higher strain rates. Steady state flow stresses as low as 700 psi have been observed at 1450°C, but only at very low strain rates. All stress, strain, and strain rate data reported from these bending experiments have been temperature corrected, i.e., adjusted for dimensional changes in specimen and loading fixture induced by thermal expansion (see Eq. 5).

Kinetics of Deformation

Plots of ln $\dot{\epsilon}$ as a function of lnσ for seven such bending experiments over the range 1450 - 1700°C are shown in Figure 8, and, for comparison, some preliminary data from McBrayer's deformation of [111] oriented single crystals and from Choi's compression tests on polycrystalline spinel are included. All show a strong stress-strain rate dependence, with the constant n always having values well above 2, with an average near 3.

In the bending experiments, it has been observed that n varies from just less than 2 to about 5 over individual sequences of the plot for a given specimen, depending upon whether the strain rate was being lowered (yielding low n values) or increased (high values). In these experiments, strain rate was normally changed abruptly by a factor of two, either halving or doubling, without unloading the specimen.

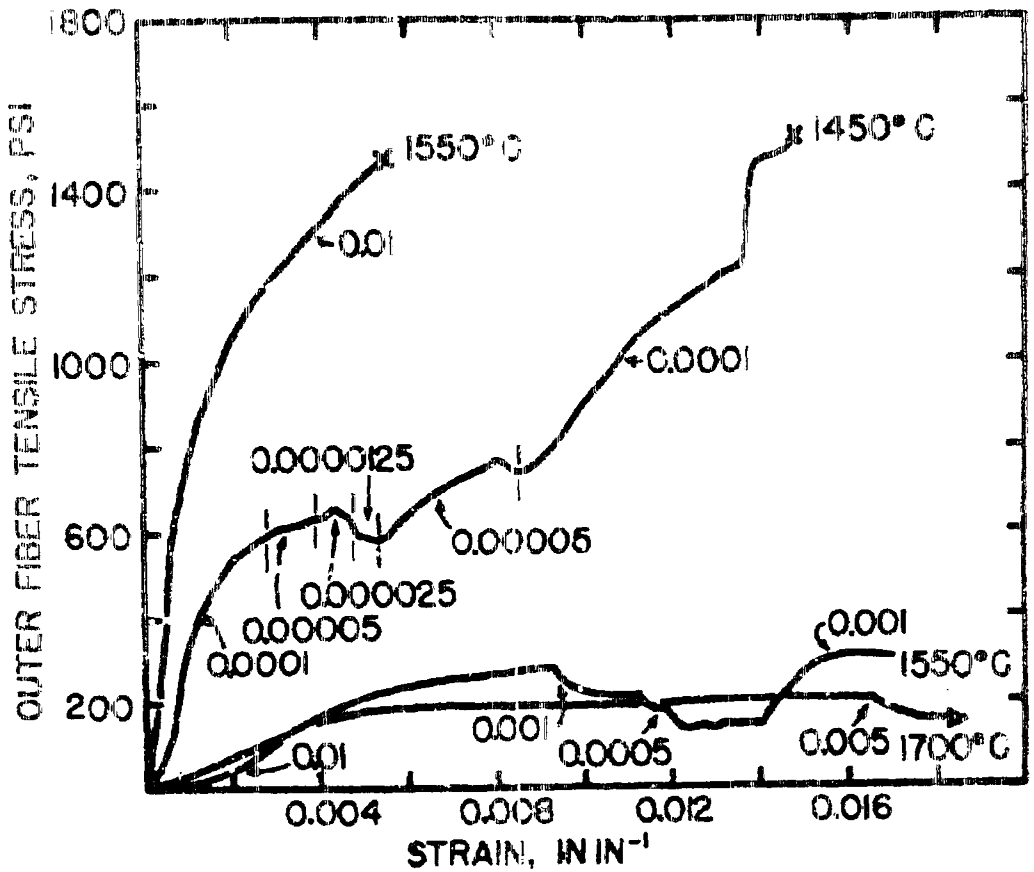

Figure 7. Stress-Strain Diagrams for Polycrystalline Spinel Deformed in Transverse Bending above 1450°C. Vertical marks denote change in strain rate; numbers indicate applicable strain rates (in. in^{-1} min^{-1}).

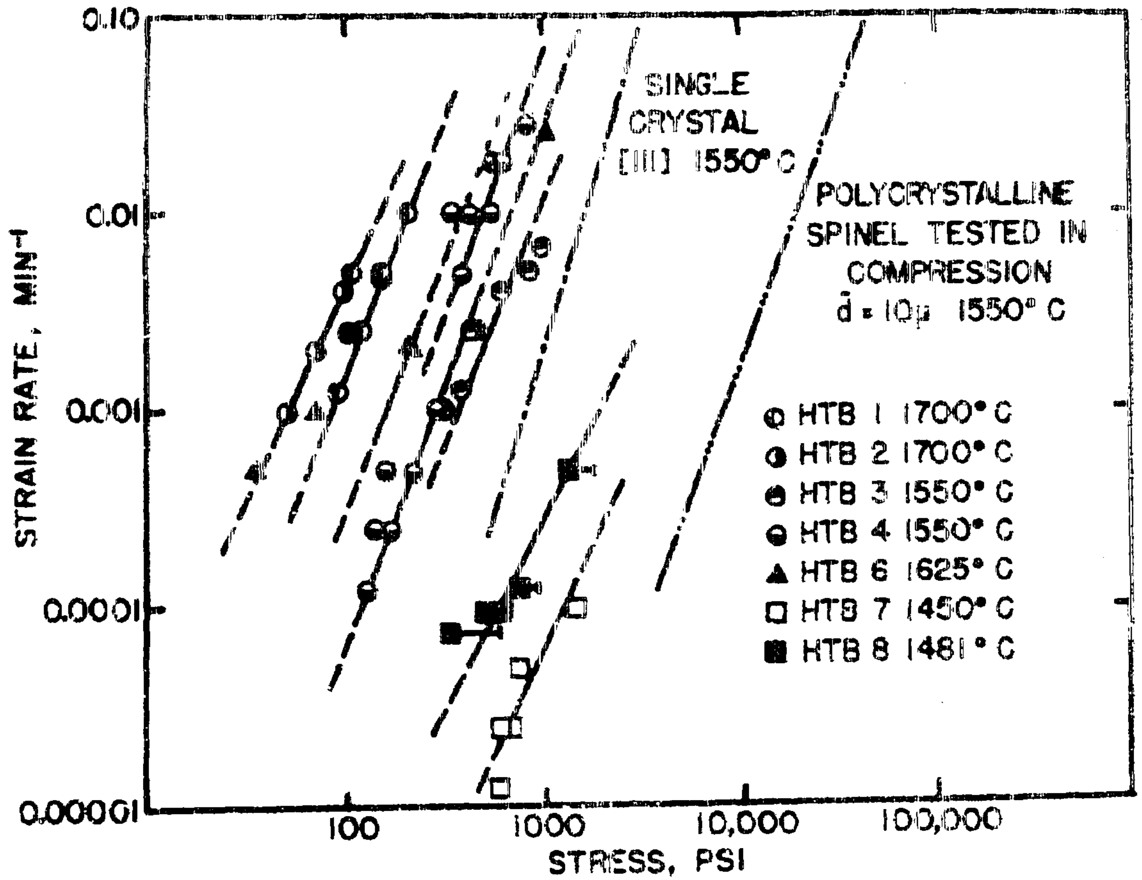

Figure 8. Logarithmic Strain Rate-Stress Plot for Polycrystalline Spinel Deformed in Bending (HTB Series). Includes comparative data for compressive flow in single crystal spinel (after McBrayer) and polycrystalline spinel (after Choi).

If a gear change was required to further extend the strain rate range, the stress generally was removed, allowing the specimen to "rest" for a period of several minutes. When testing was resumed at the higher strain rate range, the slope would be significantly lower than normal, usually over three levels of increased strain rate spanning almost a half decade. For the bending experiments, the averaged value for n is 2.7. Therefore, at constant temperature

$$\dot{\varepsilon} \approx 8.75 \times 10^{-10} \sigma^{2.7} \tag{5}$$

where σ = outer fiber tensile stress = $\dfrac{3 P a F_1}{b h^2}$

$\dot{\varepsilon}$ = outer fiber tensile strain rate (min^{-1}) = $6 h \Delta\dot{y} F_2$

a = moment length = $\dfrac{l_1 - l_2}{2}$ = 0.250"

P = load (pounds) h = height (in.)

b = breadth (in.) $\Delta\dot{y}$ = crosshead strain rate (in. min^{-1})

F_1 = thermal correction factor for stress = $\dfrac{1 + \alpha_2 \Delta T}{(1 + \alpha_1 \Delta T)^3}$

F_2 = thermal correction factor for strain = $\dfrac{1 + \alpha_1 \Delta T}{(1 + \alpha_2 \Delta T)^3}$

α_1 = coefficient of linear thermal expansion for spinel = $7.9 \times 10^{-6}\ {}^{\circ}C^{-1}$.[25]

α_2 = coefficient of linear thermal expansion for molybdenum = $5.7 \times 10^{-6}\ {}^{\circ}C^{-1}$.

ΔT = difference between test and room temperatures, $^{\circ}C$.

A plot of ln $\dot{\varepsilon}$ as a function of $\frac{1}{T}$ for three levels of flow stress in bending at temperatures of 1550°C and above is shown in Figure 9.

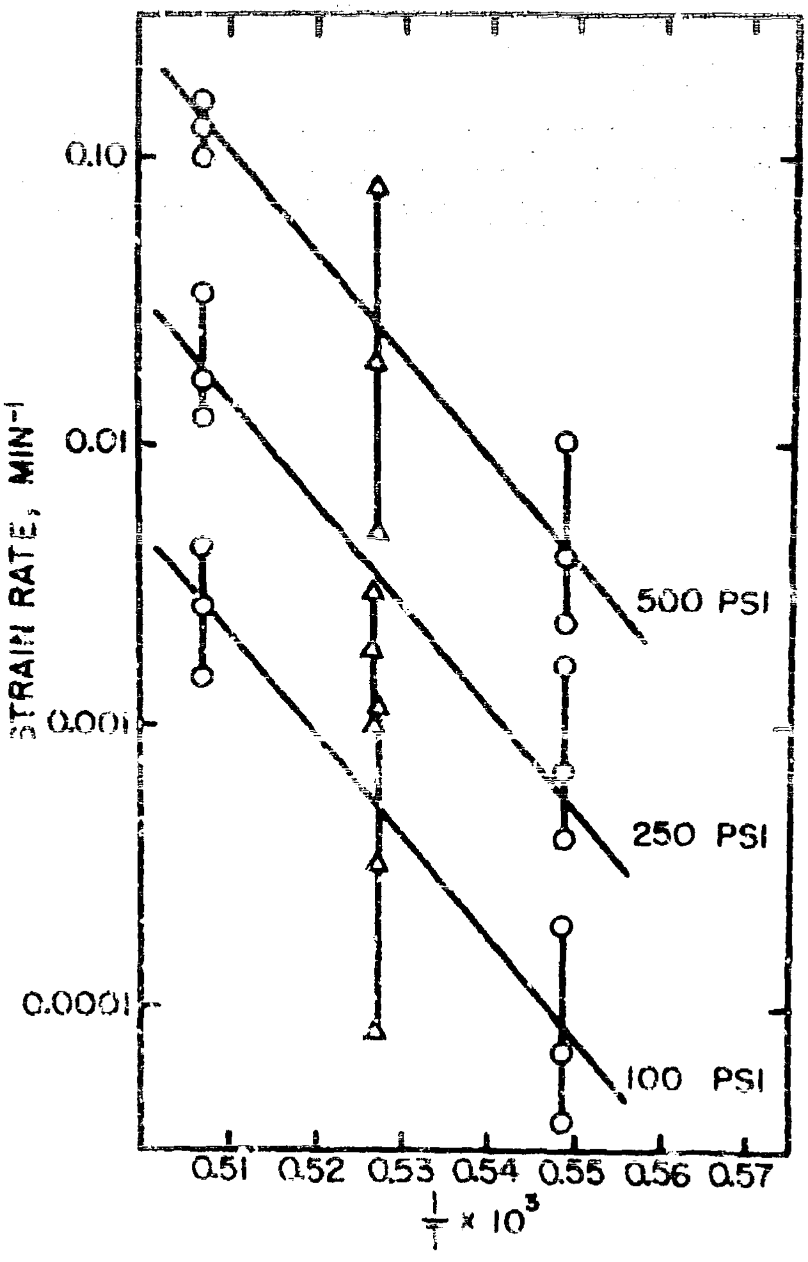

Figure 9. Semi-Logarithmic Plot of Strain Rate as a Function of Reciprocal Absolute Temperature at Applied Stresses of 100, 250, psi for polycrystalline Spinel Deformed in Bending. Midpoints represent nominal values; upper and lower points represent extreme values based on highest and lowest values of n.

Lines having one constant slope provide reasonable fits at all three levels, indicative of a reasonably well behaved relationship responsive to a single activation energy over the temperature range. Graphical evaluations of these preliminary data provide an empirical rate equation for bending of the form

$$\dot{\varepsilon} = A'\sigma^{n} e^{-Q/RT} \tag{6}$$

where, in this experiment

$$\dot{\varepsilon} = 0.375 \times 10^{15} \sigma^{2.7} e^{-214,158/RT} \tag{7}$$

The apparent activation energy, Q, has a value of approximately 214 Kcal, and the preexponential, A', has a value of approximately 0.375 x 10^{15}. These values are not inconsistent with some thermally activated process involving self-diffusion of a slow moving ionic species. This experimentally derived expression only differs in one respect, the lack of a linear $\frac{1}{T}$ term, from the Weertman[15] equation for a creep process controlled by dislocation climb

$$\dot{\varepsilon} = A \frac{\sigma^{n}}{T} e^{-Q/RT}. \tag{8}$$

Equation 7 can be adjusted to Weertman form by employing a typical experimental value of $\frac{1}{T}$ (0.55×10^{-3}) as a divisor for A', yielding

$$\dot{\varepsilon} \approx 0.68 \times 10^{18} \frac{\sigma^{2.7}}{T} e^{-214,000/RT} \tag{9}$$

for high temperature bending in spinel.

Microstructural Evidences of Deformation

Microstructural studies in polycrystalline spinel are fairly

awkward by conventional polish-and-etch techniques, partly because etching spinel in sulphuric acid at 270°C is a difficult and unpleasant business, and also because cold working of the surface during polishing usually leads to heavy scratch textures on the etched specimen. For many studies, it has been found that replicas of fractured or "free" thermally etched surfaces, when shadowed with chromium vapor, lend themselves particularly well to optical microscopy at magnifications up to about 1000X. All those illustrated here were taken of plastic film replicas* shadowed with 200 - 400 Å of Cr metal at an incident angle of approximately 45°, and were photographed on Polaroid P/N 55 film using a reflected light microscope** with bright field illumination from a zirconium arc source (with green filter).

The polycrystalline spinel tested in the bending experiments was fine grained and rather uniform in texture, with a slightly bimodal grain size distribution averaging 2 - 3μ. The matrix was quite fine with a grain size on the order of 1 - 2μ, and grains which had grown larger were only about 5μ maximum. Figure 10 illustrates the microstructure as revealed by fracturing the material at room temperature

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*Replicating Solution, a product of Ladd Industries, Inc., 159 Wagon Rd., Roslyn Heights, N. Y. The solution is thinned with acetone to optimum fluidity; after drying the replica is peeled off with transparent tape.

**Model MeF Universal Microscope, a product of Optische Werke C. Reichert, Vienna.

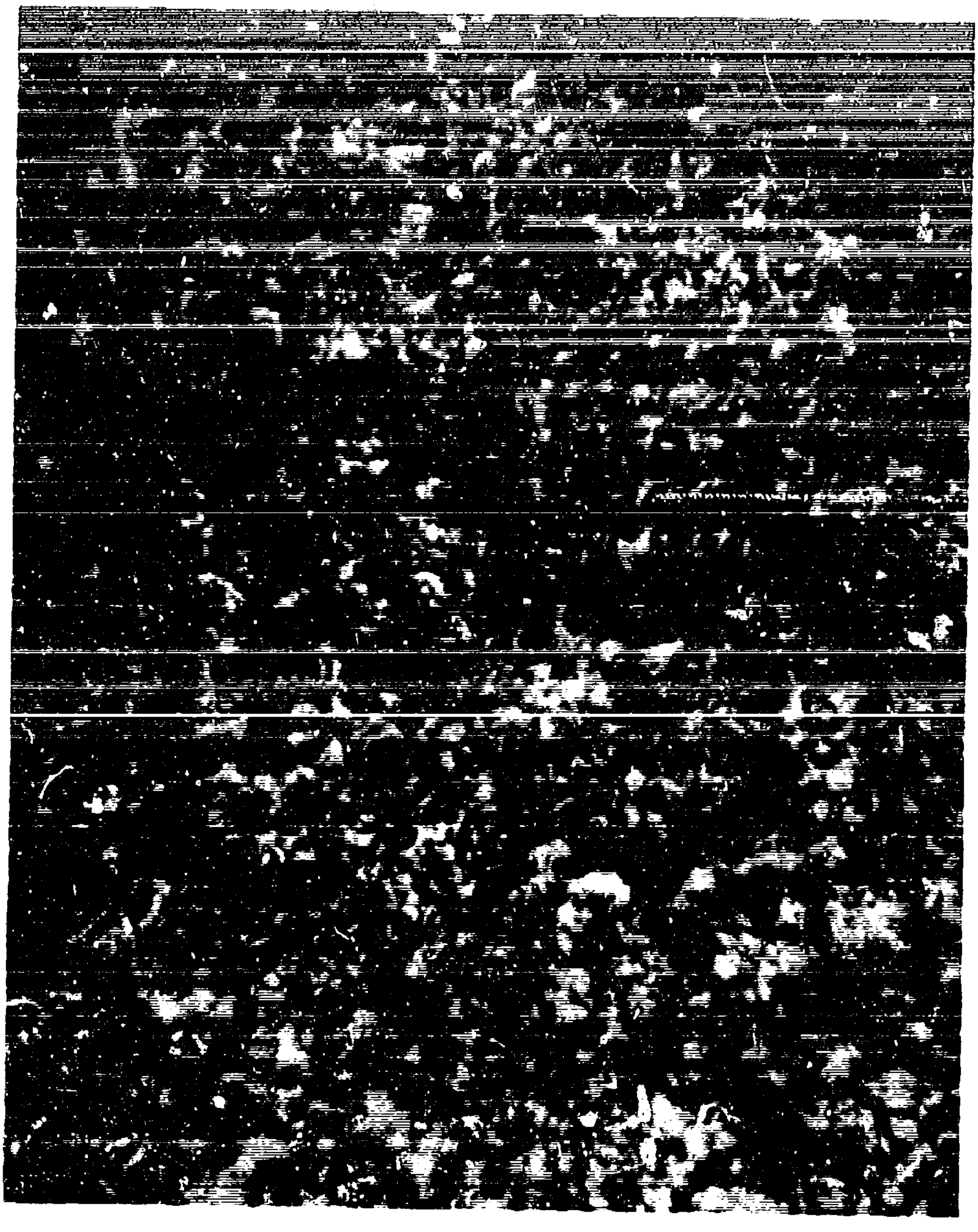

Figure 10. Microstructure of Polycrystalline Spinel Prior to High Temperature Deformation. Cr-shadowed replication fractograph; specimen fractured in bending at room temperature. X2200.

in bending. Some evidence of the pore phase (2.6%) can be noted, and both trans-and-intergranular fracture paths are observed.

Some grain growth occurred at test temperature, as shown in Figure 11, replicated from a specimen deformed to about 4% strain prior to deliberate fracture at higher strain rates at 1550°C. The lower edge of the cross section in this Figure was the trace of the tension face of the bent specimen. The matrix grains were only slightly larger than those in unheated spinel, but the larger grained fraction had grown to 10 - 25 μ size, the example shown being one of the largest observed. These larger grains frequently showed evidence of intersecting slip, whereas the finer matrix grains did not, at least within the resolution of the optical microscope. Although this region had been subjected to considerable strain (normal to the picture plane) prior to fracture, there was no evidence of the development of grain boundary cracks. Finally, it should be noted that fracture was partly intergranular, partly transgranular.

Replicas taken from thermally etched tension and compression surfaces confirmed the absence of intergranular cracks after extensive deformation. Many of the larger grains showed evidences of slip bands and the onset of wavy slip indicative of plastic deformation. Two such examples taken from the tension surface of a specimen deformed at 1700°C to an outer fiber tensile strain of almost 0.05 in. in^{-1} are shown in Figure 12.

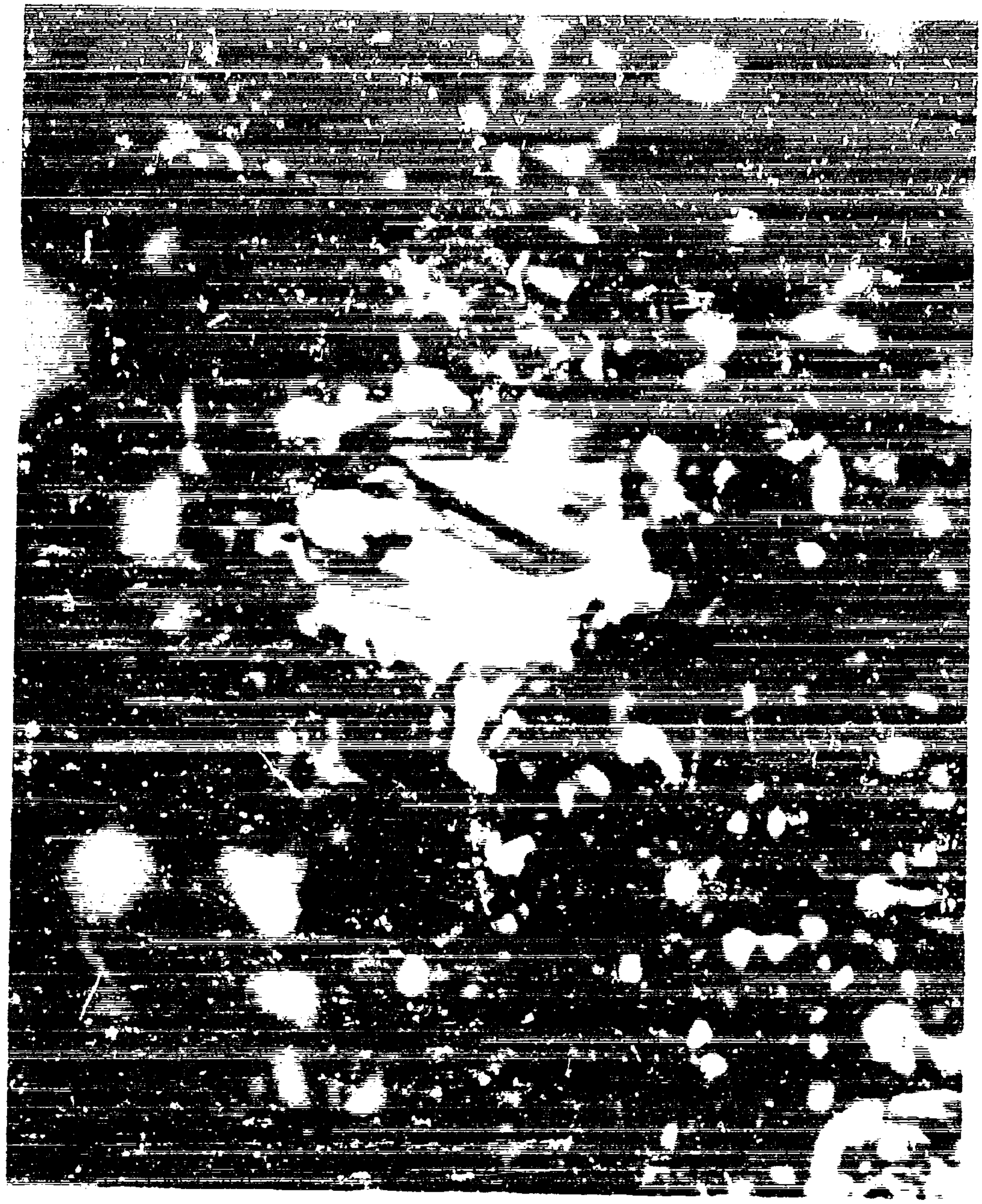

Figure 11. Microstructure of Polycrystalline Spinel After Deformation at 1550°C. Transverse section near tension surface, Cr-shadowed replication fractograph; specimen strained approximately 3%, then fractured at higher strain rate. X2200.

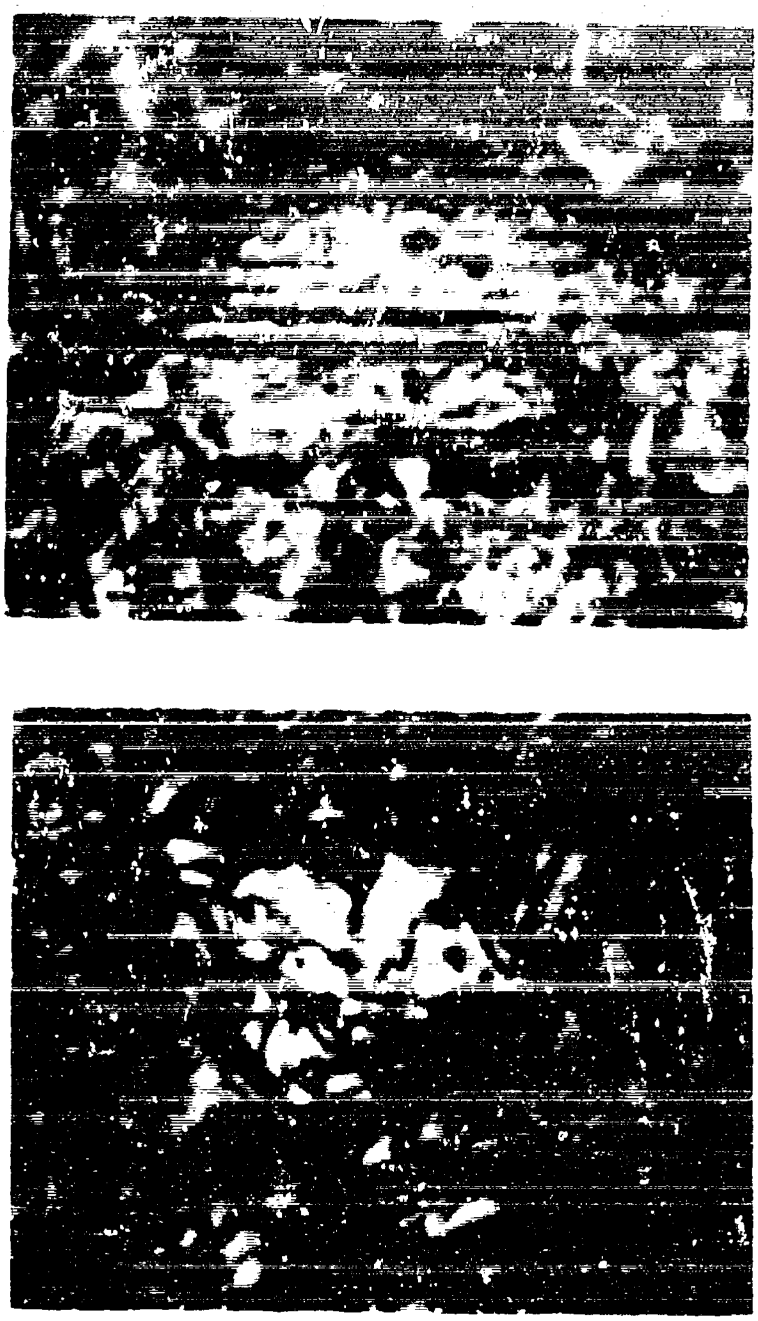

Figure 12. Microstructure of Tension Surface of Polycrystalline Spinel Bent at 1700°C. (a) Slip bands developed in large grain; (b) wavy slip developed in large grain with approximately [111] orientation. Strain direction horizontal. Cr-shadowed replica of ground surface thermally etched during test. X2200.

Discussion

One can categorically rule out grain boundary sliding as the rate-controlling mechanism in bending of spinel above 1550°C on the grounds that the boundaries were still intact after as much as five percent strain, and because a viscous stress-strain rate dependence (n = 1) was not observed. Nabarro-Herring diffusional creep is also considered quite unlikely on the grounds that the observed strain-rate-stress coefficient was much higher than unity, and also because strain rates employed in these bending and compression experiments exceeded those normally observed at comparable temperatures for diffusional creep in ceramics[7] by at least two orders of magnitude.

Stress-strain relationships, including work hardening, and the kinetic analyses, provided strong support for a plastic deformation process involving hardening by dislocation interactions with steady state flow being governed by some counteracting thermally activated recovery process. These preliminary data obtained from a relatively small number of specimens are not considered adequate for exact quantitative determinations of the values of n or Q. The values reported are adequate to demonstrate plasticity, but n is considered presently to be somewhat too low, and Q somewhat too high. Choi's compressive studies, based on many specimens, indicate that the nominal value of n for polycrystalline spinel may be as high as 4 or 5, and that Q may be as low as 165 Kcal.

No decrease in bulk density was observed, in fact, density was

increased during bending. Microstructural evidence also confirmed the retention of structural integrity after extensive deformation in bending, and provided many examples of plastically altered grains. Other micrographs have indicated possible reorientation by polygonization near grain boundaries in coarse grained spinel deformed in compression. Such observations are considered to give some preference to the Weertman concept of dislocation climb as the rate-controlling mechanism for plastic flow in spinel, although the kinetic data now available are not complete enough to permit an unambiguous designation of the exact mechanism.

CONCLUSION

Spinel of high purity and fine grain size is a ductile ceramic when deformed by bending at high temperatures. Above 1550°C, it displays strain rate sensitivity, strain hardening, recovery, extensibility, and other plastic traits one normally associates with a face centered metal. Under these conditions, it is not brittle in bending unless the strain rate is high, well in excess of 0.01 min^{-1}. However, the onset of plasticity in the 1450°C temperature range reduces useful strength to a relatively low level, not because of fracture, but because of rapid creep. This factor should be taken into account in any engineering application of pure fine grained spinel at these temperatures.

The availability of a reasonably ductile polycrystalline ceramic

at experimentally- and industrially-attainable temperatures does suggest some interesting possibilities for future investigation. One can predict with real certainty that spinel will continue to be attractive as a model material for research studies concerned with hot working and strengthening of high temperature materials.

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